IS 12292 (1988): Lead sub oxides (lead oxide) for lead-acid storage battery [CHD 1: Inorganic Chemicals]

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Indian Standard

SPECIFICATION FOR LEAD SUBOXIDE (LEAD OXIDE) FOR LEAD-ACID STORAGE BATTERY

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Gr 2

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Indian Standard

SPECIFICATION FOR LEAD SUBOXIDE (LEAD OXIDE) FOR LEAD-ACID STORAGE BATTERY

0. FOREWORD

0.1 This Indian Standard was adopted by the Bureau of Indian Standards on 15 February 1988, after the draft finalized by the Inorganic Chemicals (Misc) Sectional Committe had been approved by the Chemical Division Council.

0.2 Lead suboxide or lead oxides find use in the manufacture of lead acid storage battery paste which is usually prepared by mixing them with sulphuric acid and distilled water along with some other minor substances to improve the binding characteristic to the grid frame into which it is held. This is the active material which delivers the electrical energy reacting with the electrolyte sulphuric acid during discharge, and by charging process, direct current is passed to convert the lead sulphate which is the product of discharge to lead dioxide on the positive and spongy lead on the negative plates as the reversible reaction.

0.3 Lead suboxide is a mixture of lead oxide (PbO) and free lead (Pb). It can be classified according to the method of manufacture involving various proportions of PbO and Pb. Lead suboxide is used the manufacture of lead acid storage battery. The existing Indian Standards on red lead and litharge include IS : 57-1965* and IS : 53-1975† which are mainly concerned with the paint manufacture.

0.4 Lead suboxides are manufactured in Barton Pot where air is passed through the molten lead and the oxide is then led through classifier and cyclone and is collected as lead suboxide known as Barton oxide. In the other process, the lead is cast in balls or cut into pieces and fed in a ball mill where by attrition between the balls, the oxide is produced which is further classified and collected as lead suboxide. Both the processes produce lead suboxide which is used in lead acid battery. They differ in physical properties like particle size and shape but most of the other properties remain same. The ultimate end use of both the suboxides are alike. The particle shape of mill oxide is leafy and that of pot oxide is spherical. The crystal structure of these oxides are generally tetragonal and ortho-rhombic in small amounts.

0.5 The material has a tendency to oxidize and properties change when exposed to air or moisture. It is, therefore, advised to test all the requirements of Table 1 immediately after opening the sealed packing. The exact shelf life data is not available at present but investigations are in progress and this shall be incorporated as and when data is available.

0.6 For the purpose of deciding whether a particular requirement of the standard is complied with the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Specification for red lead for paints and jointing purposes (*revised*).

†Specification for litharge (*first revision*).

*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for lead suboxide (lead oxide) for lead-acid storage battery.

2. REQUIREMENTS

2.1 Description — The material shall be in fine powder form, free flowing and shall be free from lumps and foreign matter. The colour of mill oxide shall be generally grey or greenish grey; the colour of pot oxide shall be generally brownish grey tending towards yellowish grey.

2.2 The material when tested as prescribed in Appendix A shall also comply with the requirements given in Table 1. Reference to relevant clauses of Appendix A is given in col. 5 of the table.

3. CLASSIFICATION

3.1 The material shall be of the following two types:

a) Mill oxide (ball mill oxide), and

b) Pot oxide (Barton Pot).

TABLE 1 REQUIREMENTS FOR LEAD SUBOXIDE (LEAD OXIDE) FOR LEAD—ACID STORAGE BATTERY

(*Clauses* 0.5 *and* 2.2)

Sl No.	Characteristic	Requirement		Method of Test (Ref to Cl No in Appendix A)
		Mill Oxide	Pot Oxide	
(1)	(2)	(3)	(4)	(5)
i)	Apparent (Scott) density, g/ml	1·2-1·3	1·4-2·0	A-2
ii)	Residue on 63-micron IS sieve, percent by mass, *Max*	10·0	3·0	A-3
iii)	Acid absorption, mg/g, *Min*	170	100	A-4
iv)	Water absorption, ml/100 g	9-15	9-15	A-5
v)	Free metallic lead, percent by mass	20-40	20-40	A-6

3.1.1 Specification of impurities and free lead in lead suboxide shall be as agreed to between the supplier and the purchaser.

A typical limiting percentage of impurities in lead suboxide is shown below:

Element	*Maximum Percentage*
Iron	0·002
Antimony	0·001
Arsenic	0·0001
Bismuth	0·030
Copper	0·002
Silver	0·001
Tin	0·001
Zinc	0·001
Nickel	0·0001

Sum of all these impurities may not exceed 0·04 percent, when analysed in accordance with IS : 403-1964* or by spectro-photometric method as mutually agreed between the supplier and the purchaser.

4. PACKING AND MARKING

4.1 Packing — The material shall be packed in air-tight steel drums of 50 kg capacity with an opening large enough to allow the contents to be easily removed.

4.2 Marking — The container shall be marked legibly and indelibly with the following information:

a) The name of material and its net mass;

b) Type of oxide;

c) Name of manufacturer and/or recognized trade-mark, if any; and

d) Lot number and month of manufacture.

4.3 The containers may also be marked with the Standard Mark.

Note—The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5. SAMPLING AND CRITERIA FOR CONFORMITY

5.1 Lot — In any consignment, all the drums of lead suboxide of same type and manufactured in a single batch shall constitute a lot.

5.2 Samples shall be tested for each lot for ascertaining conformity of the material to the requirements of this specification.

5.3 The number of drums to be chosen from the lot shall depend upon the size of the lot and shall be in accordance with Table 2.

TABLE 2 NUMBER OF DRUMS TO BE SELECTED

Lot Size	Sample Size
(1)	(2)
Up to 25	3
26 to 50	4
51 to 100	5
101 to 150	7
151 to 300	10

5.4 The drums shall be selected from the lot at random and in order to ensure the randomness of selection, procedure given in IS : 4905-1968* may be followed.

5.5 Number of Tests—Tests for determination of all the characteristics shall be conducted on composite sample by taking equal amount of lead suboxide from each drum.

5.6 Criteria for Conformity—The lot shall be declared as conforming to the requirements of this standard if all the characteristics tested on composite sample meet the relevant requirements given in Table 1.

*Methods of chemical analysis of lead and antimonial lead (*revised*).

*Methods for random sampling.

APPENDIX A

(*Clause* 2.2)

METHODS OF TEST FOR LEAD SUBOXIDE (LEAD OXIDE) FOR LEAD-ACID STORAGE BATTERY

A-1. QUALITY OF REAGENTS

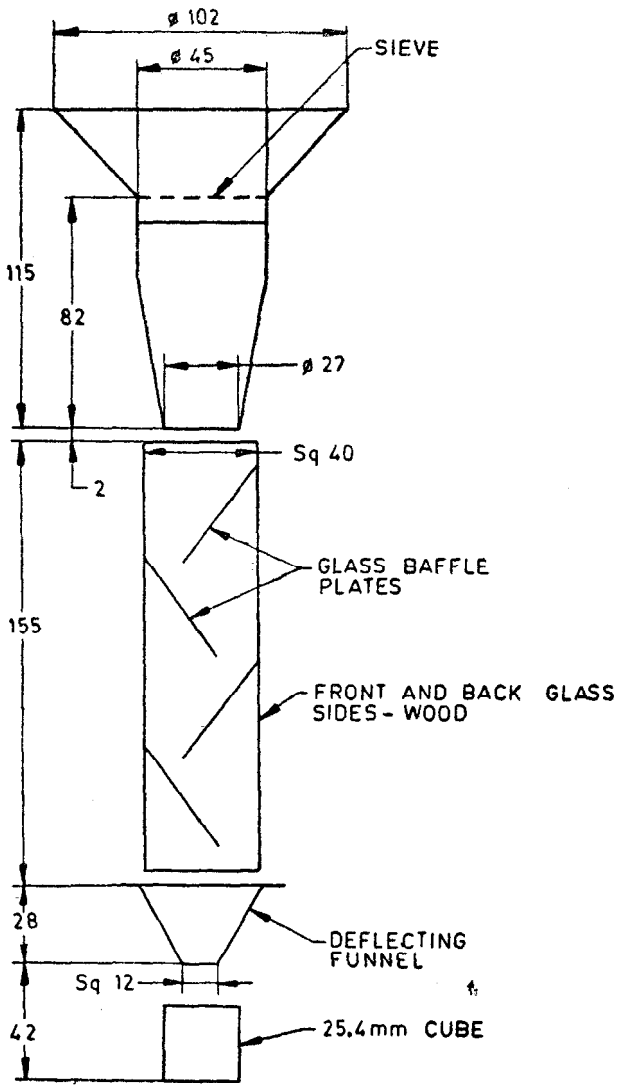
A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be used in tests.

NOTE—'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. APPARENT (SCOTT) DENSITY

A-2.1 Apparatus

A-2.1.1 *Scott Volumeter*—The apparatus (*see* Fig. 1) consists of a hopper, across the bottom of which is stretched a 180-micron stainless steel sieve, a funnel for directing pigment into a box contain a series of glass baffles and a funnel at the bottom to direct the pigment into a cubic receiver of 25·4 mm side. Pigment passing through this device is always loaded into the receiver under uniform conditions.



All dimensions in millimetres.

FIG. 1 SCOTT VOLUMETER

A-2.1.2 *Pallet Knife*

A-2.2 Procedure—The powder under test is placed inside hopper and slowly brushed, when the powder gradually falls into the weighed empty cube after passing through a series of glass baffles. This process is continued till the cube fills up into a cone above the top of the cube. The surplus powder is struck off the cube with the knife and the cube lightly tapped. The final mass of the cube is then taken.

A-2.3 Calculation

$$\text{Apparant density, g/ml} = \frac{(M_2 - M_1)}{16{\cdot}40}$$

where

M_2 = mass in g of cube + oxide, and

M_1 = mass in g of empty cube.

A-3. RESIDUE ON 63-MICRON IS SIEVE

A-3.1 Procedure—Weigh accurately 10 g of lead suboxide and place it on the sieve and gently rub with wide hair brush till all the fine particles pass through the sieve. Collect the coarse particles and weigh.

A-3.2 Calculation

$$\text{Residue on sieve, percent by mass} = 10 \times M_1$$

where

M_1 = mass in g of the material retained on the sieve.

A-4. ACID ABSORPTION

A-4.1 Reagents

A-4.1.1 *Dilute Sulphuric Acid*—15 percent (*v/v*).

A-4.1.2 *Standard Potassium Hydroxide*—1 N.

A-4.1.3 *Phenolphthalein Indicator Solution*—Dissolve 0·1 g of the indicator in 100 ml of 50 percent rectified spirit.

A-4.2 Procedure—Place 100 ml of sulphuric acid solution in a 250-ml wide mouthed conical flask. Weight 50 g of suboxide and introduce it slowly to the solution. Cap the bottle tightly and rotate it end to end at 18 rpm for 10 minutes. The experiment is carried out at room temperature. No measures are taken to regulate the temperature of the mixture. Therefore, at the end of mixing, the temperature in the bottle

*Specification for water for general laboratory use (*second revision*).

will be higher than that of ambient temperature. Let the mixture stand for 5 minutes. Decant about 40 ml of the supernatant liquid and filter it through dry filter paper. Let the filtrate cool down to ambient temperature and pipette 25 ml of it for titration with standard 1 N potassium hydroxide solution. Carry out a blank with same quantity of chemicals and reagents as described above.

A-4.3 Calculation

Acid absorption, mg/g

$$= \frac{10 \times (A-B) \times N \times 9}{20}$$

where

A = Volume in ml of standard potassium hydroxide solution consumed by the sample,

B = Volume in ml of standard potassium hydroxide solution consumed by the blank, and

N = normality of standard potassium hydroxide solution.

A-5. WATER ABSORPTION

A-5.1 Procedure — Place 100 g of suboxide in a dry beaker and add 7 ml of water from a burette. Mix well with a knife spatula and continue adding water in suitable increments, mixing with a chopping and stirring action and breaking up any lumps that form. Continue the water addition and mixing until the mass forms a single ball on stirring, the ball being capable of being cut up and reformed easily. The whole procedure should be completed in about three minutes.

A-5.2 Calculation — The volume of water added is reported directly as the water absorption figure.

A-6. FREE METALLIC LEAD

A-6.0 Two methods have been prescribed, namely volumetric and gravimetric methods. Gravimetric method shall be regarded as the referee method.

A-6.1 Volumetric Method

A-6.1.1 *Reagents*

A-6.1.1.1 *Disodium ethylenediamine tetraacetate solution (EDTA)*—Dissolve 45 g of reagent in 1 litre of water.

A-6.1.1.1.1 *Standardization of EDTA*—Weigh accurately 1 g of litharge and treat as in the procedure (**A-6.1.2**). From the titration, calculate the equivalent factor.

A-6.1.1.2 *Extraction solution*—Dilute 20 ml of glacial acetic acid to 1 litre with water. Add 5 g hydroxylamine hydrochloride and heat to boiling. Boil the solution for 5 minutes, cool and transfer to stock bottle.

A-6.1.1.3 *Xylenol orange solution*—0·2 percent aqueous solution, freshly prepared]

A-6.1.1.4 *Hexamine*

A-6.1.2 *Procedure*—Weigh accurately 1 g of the sample and transfer to a 250-ml conical beaker. Add 100 ml of the extraction solution and boil the contents of the beaker for 3 minutes. Remove from the hot plate, add 3 g hexamine (*see* Note) and stir gently until dissolved. Add 3 drops of xylenol orange solution and titrate with EDTA solution through the colour change red to clear lemon yellow.

NOTE—The *p*H shall be 4·5 - 5·0 after hexamine addition. If *p*H is less than 4·5, adjust by addition of excess hexamine in 0·5 g portions.

A-6.1.3 *Calculation*

Free metallic lead, percent by mass = Titre (ml) × factor × 100.

The 'factor' used in calculation shall be the equivalent factor as given in **A-6.1.1.1.1.**

A-6.2 Gravimetric Method

A-6.2.1 *Procedure*—Weigh 2 g of lead suboxide accurately and dissolve in 10 percent dilute acetic acid until only free lead remains in the beaker. Filter the solution along with free lead using two Whatman filter papers (No. 41) together. Wash thoroughly to remove the lead acetate solution. Rinse with alcohol. Dry the free lead in an oven, preferably in a vaccum oven at a temperature of 100°C. Weigh the free lead with filter paper, using the other filter paper as the counter pore.

A-6.2.1.1 Express the mass of lead as the percentage of lead suboxide taken.

A-6.2.2 Alternatively, metallic lead content can also be determined using mannitol solution in place of dilute acetic acid.

A-6.2.2.1 *Procedure*—To 1 - 2 g of weighed, finely divided specimen placed in 250-ml beaker, 100 ml of mannitol solution (*see* Note) is added. The mixture is heated and held at boiling temperature for 5 minutes. Filter the mixture with a pre-weighed sintered glass crucible of medium pore size. Wash lead residue five times with hot water, three times with acetone and finally once with ether. Dry the crucible under vacuum. Weigh the crucible an hour later.

NOTE—For preparing mannitol solution, dissolve 100 g of sodium hydroxide, 20 g mannitol and 10 g of hydrazine dihydrochloride in water and make up to 1 litre.

A-6.2.2.2 *Calculation*

$$\text{Free metallic lead, percent by mass} = \frac{\text{Mass in g of free metallic lead} \times 100}{\text{Mass in g of sample taken}}$$

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